Sandia's μChemLab™ in Industrial Applications

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Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, For the United States Department of Energy under contract DE-AC04-94AL85000.

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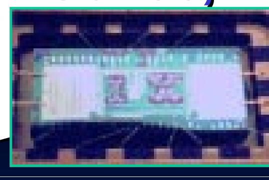
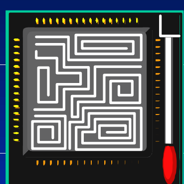
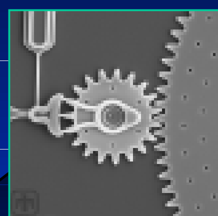
Sandia's Enabling Capabilities Produce Miniature Sensors, Processors, and Communication Systems

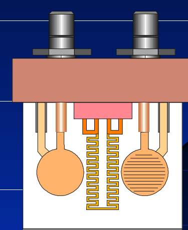
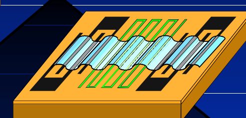
Sense, Process, Communicate



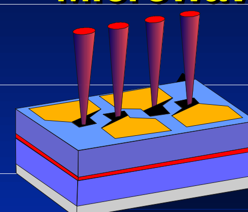
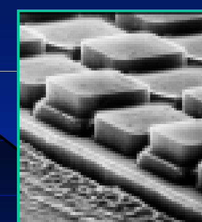

Micromachines, Microelectronics

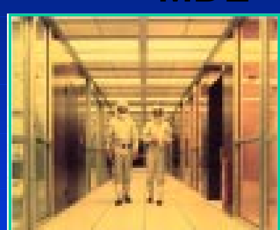
Microsensors

Photonics, Microwave Circuits

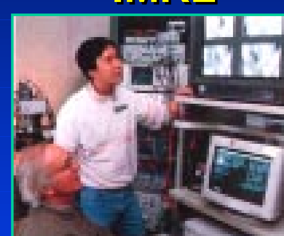



Microelectronics Development Laboratory MDL



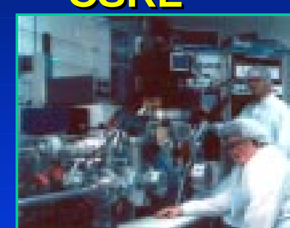
Over 30,000 ft^2 of clean room, 0.6 μm CMOS Fabrication Facility

Integrated Materials Research Laboratory IMRL



Materials Fabrication and Characterization, including Plasma Deposition and Surface and Interface State Characterization

Compound Semiconductor Research Laboratory CSRL



MOCVD, MBE Deposition, Electron Beam Lithography, Reactive Ion Beam Etching

µChemLab Application Strategy

Sensitive
Selective
Fast

Low Power
Hand Held
Low Cost
Versatile

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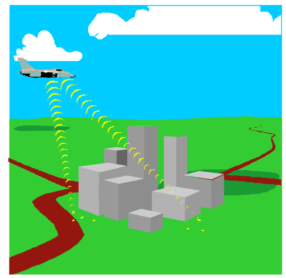
Non-proliferation



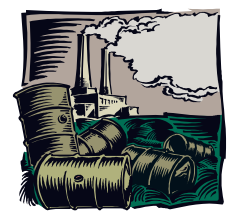
Counter Terrorism

Military (CW/BW)

Biomedical Diagnostics

Industrial Processes

Environmental



Industrial Hygiene

Food and Water Safety

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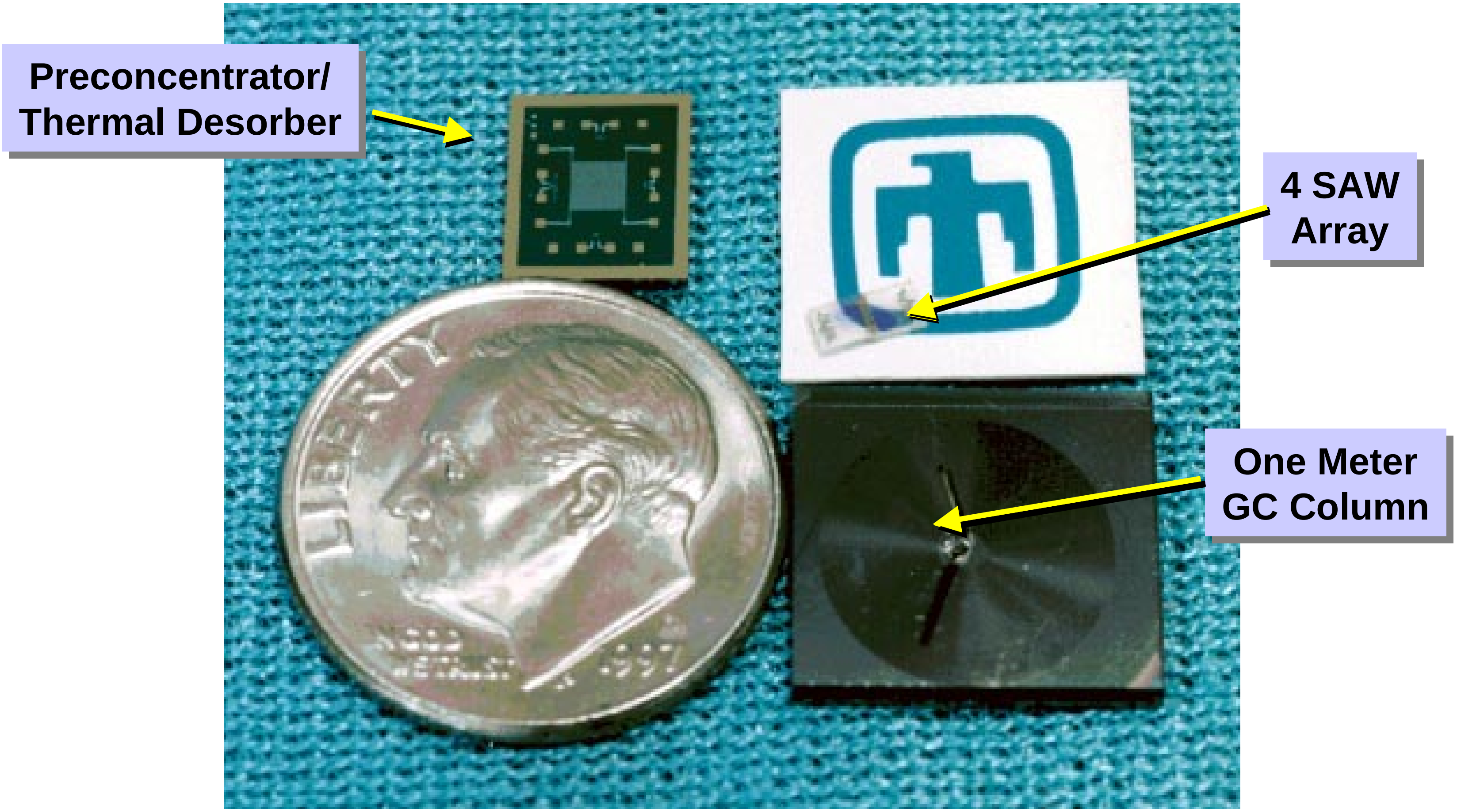
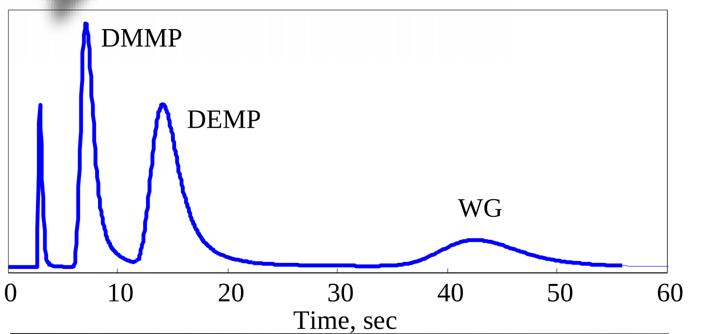
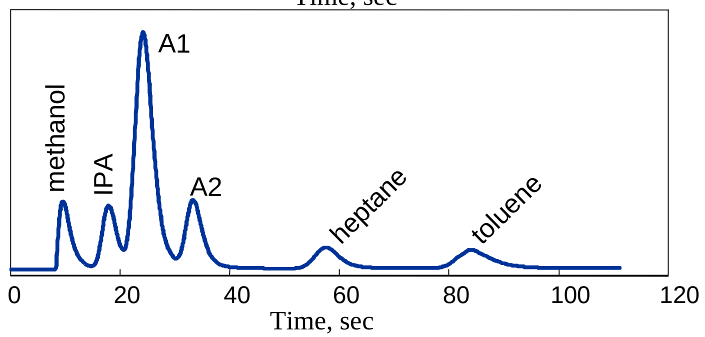
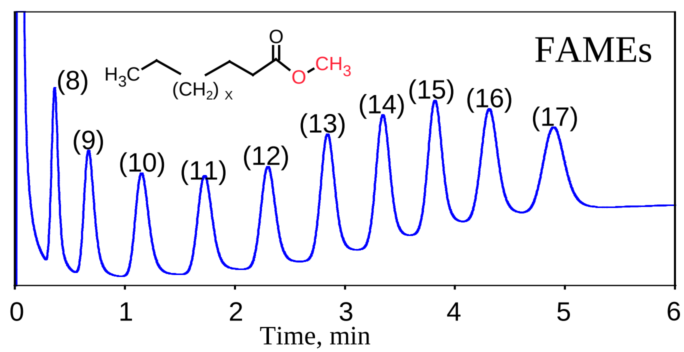
Microfabricated Components for Sandia's µChemLab



Broad Applicability







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µColumns Tailored to Specific Applications

- Chemical warfare agents/simulants
- Pharmaceutical drying, VOCs
- Biological analysis, FAMEs
- Light gases: CO, CO_2, C_1-C_4

Specificity Achieved Through

- Specific wall coatings or packings
- Column length
- Rapid temperature cycling

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Ethylene Refinery

Need quantitative measurements of:

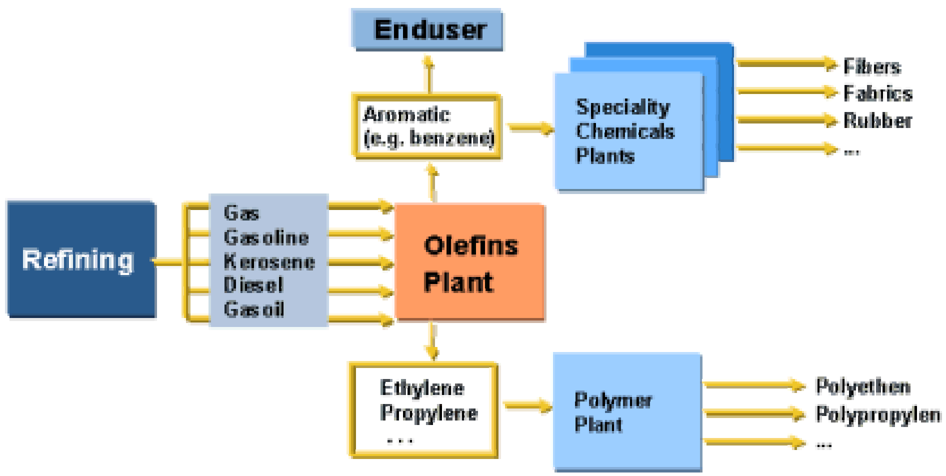
- ethane, ethylene, acetylene, carbon monoxide
  also present: carbon dioxide, methane

Changes from Sandia gas phase μChemLab:

- High, positive pressure
- Diaphragm micro-injection valve
- PDID (pulsed discharge ionization detector)
- permanent installation in hazardous environment

Current OIT Project

Petroleum Industry: Ethylene Production



Problem Statement:

- Ethylene: high-volume domestic and worldwide production
- 1999 US production ~26 M ton
- Production via batch dehydrogenation of ethane feedstock
- Acetylene is undesirable byproduct

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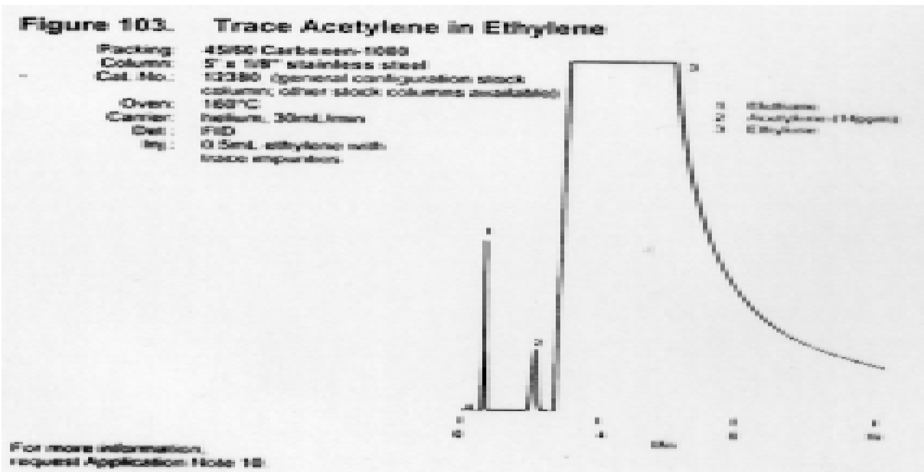
Issues

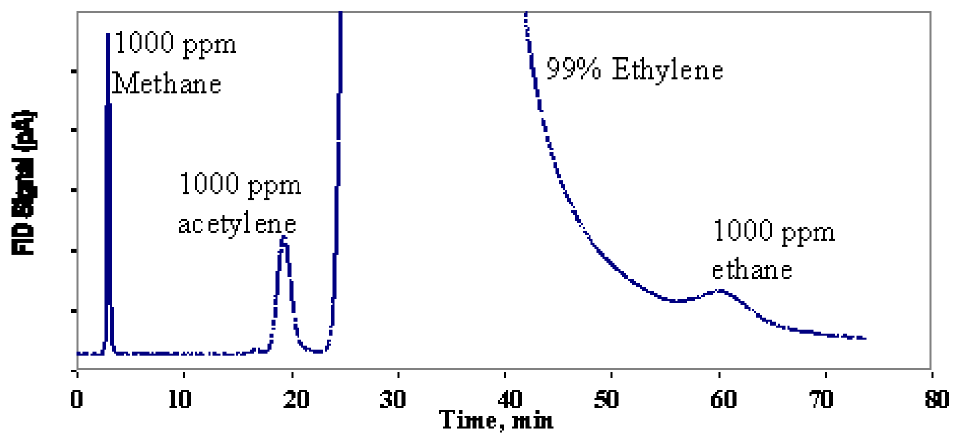
- Acetylene content controlled by catalyzed reaction in ARU requiring rapid process feedback control
- Disrupt conditions in ARU occur on short (< 3min) time scales
- Reactor runaway requires dumping to flare ($40K/hour)
- Current process methods are expensive to implement and maintain

Goals

- Reduce product loss
- Increase yield efficiency
- Reduce overall energy consumption
- Lower instrumentation costs

Advantages of Integrated Microsystem





- Replace costly instruments ($30K/each) in expensive shelters ($300K/each) with low-cost, MEMS technology
- Small-footprint, on-line process monitoring (intrinsically safe)
- Faster (1 min cycle time) process monitoring
- Improve real-time process control for enhanced process energy efficiency
- Allow increased sensor density for ARU stages

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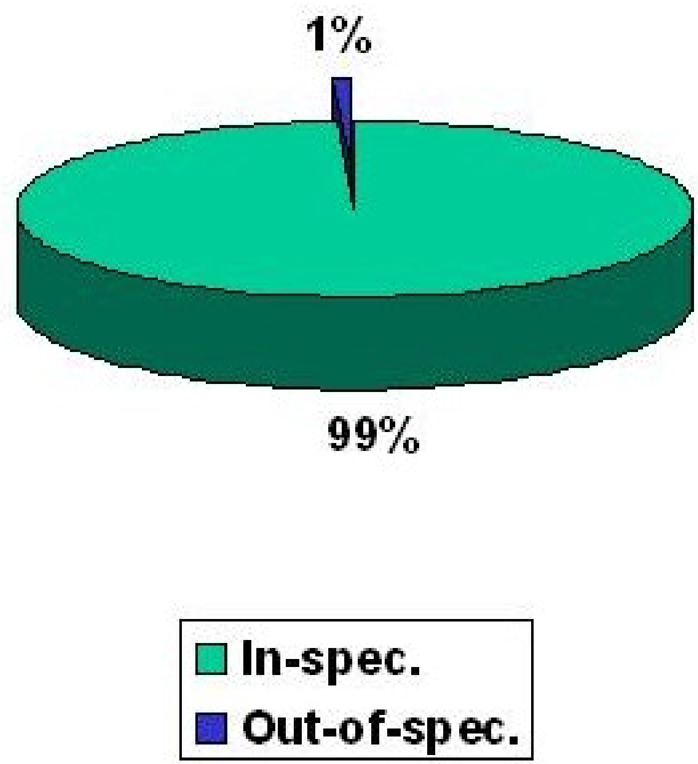
Estimated Savings

- Faster cycle-time analyzers will reduce product loss from 12.6 M lbs. to 2.5 M lbs.
- Equivalent to $400K/year/ARU product cost + energy efficiency savings

Present Ethylene Product Yield *



*Data from a Phillips ARU over a 16-mo. interval

Budget

DOE-OIT	FY 2001	FY 2002	FY2003
Sandia National Labs	$300K	$325K	$325K

Industry in-kind			
Thermo-ONIX	$275K	$275K	$225K
Phillips Petroleum	$25K	$50K	$100K

Total				3 Year Total
	$600K	$650K	$650K	$1900K

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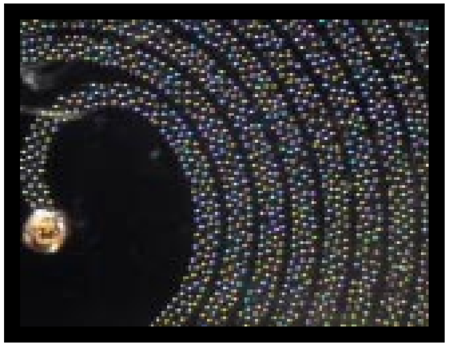
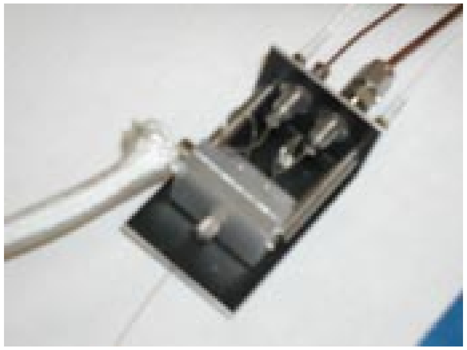
Technical Accomplishments

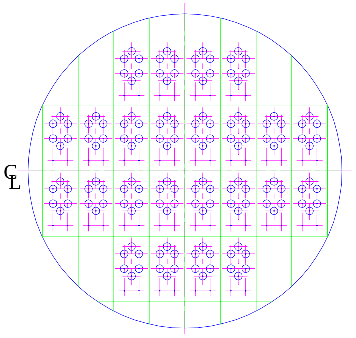
- Demonstrated success of micro-packed column to separate gases of interest
  – 20 cm long channel; 300 x 300 μm cross section; packed with Supelco carboxen 1000E, 75-100 μm





- Identified micro-PDID detector as best candidate for system
  – evaluation in progress

- Completed micro-valve design
  – preliminary fabrication in progress



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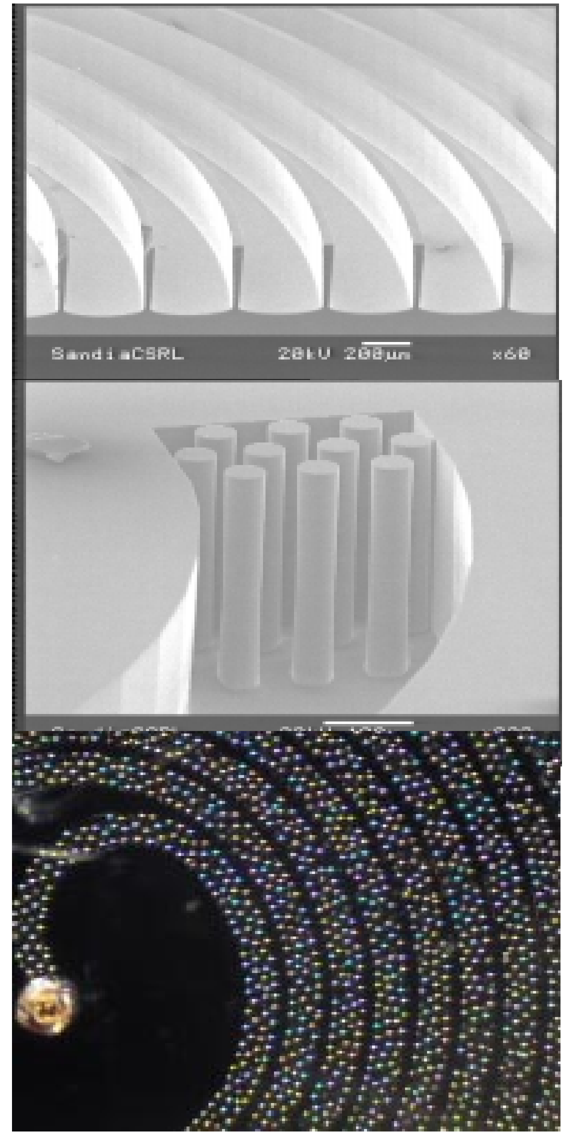
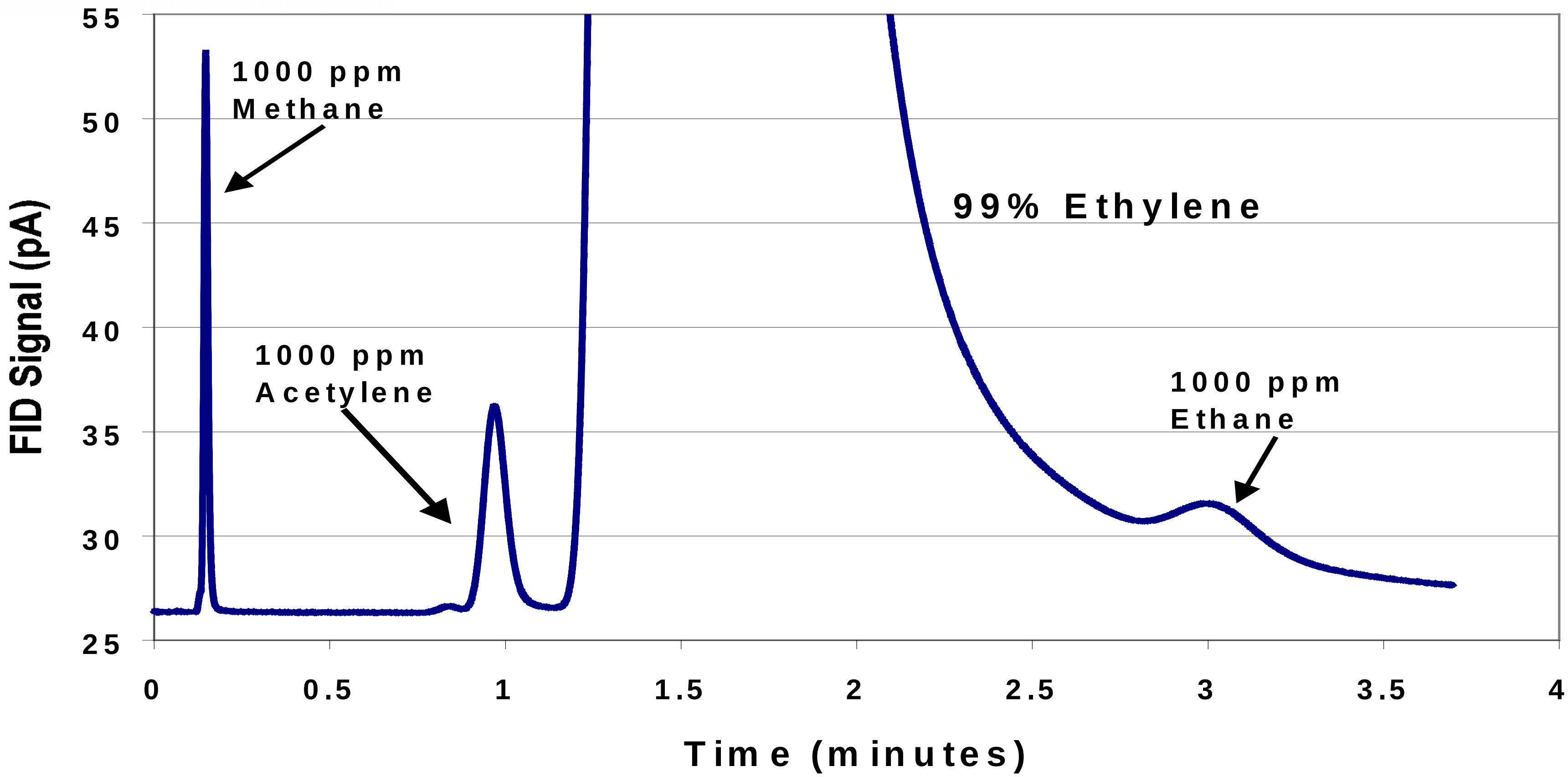
On-Chip, Packable Micro Column

- Enables use of existing column packing materials and technology
- Allows necessary retention of light molecules for adequate separations
- Improves resolution
- Patent filed

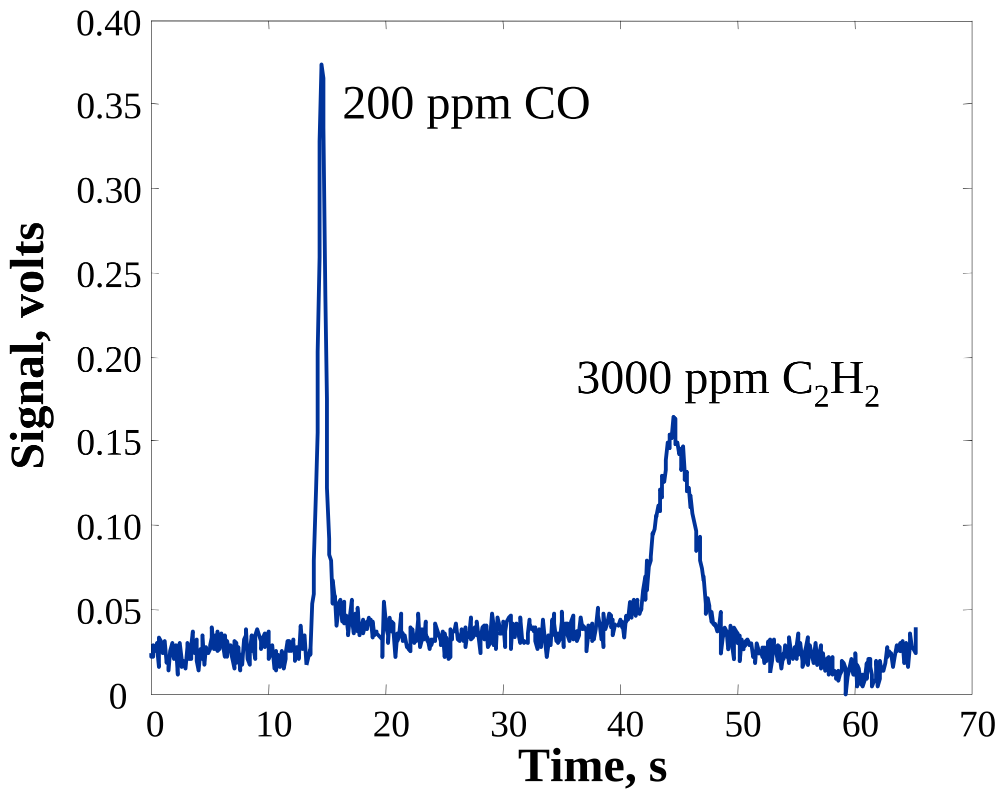


Micropacked Carbon Column Effectively Separates Acetylene in Ethylene



10 µL injection; 60 °C isothermal; 10 p.s.i.g. N_2 Supelco carboxen 1000E packing

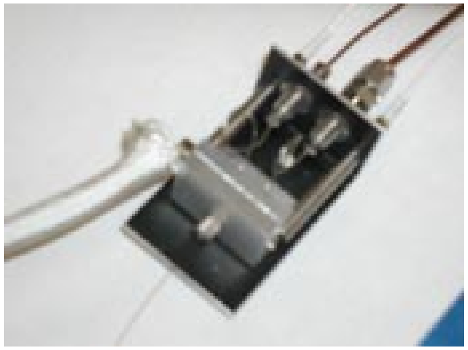
Valco micro Pulsed Discharge Ionization Detector



commercial diaphragm valve/µGC/PDID

Advantages:

- Allows CO, CO_2 detection
- Flame-proofing not required
  - instrument located at-line
- Sensitive
- Robust



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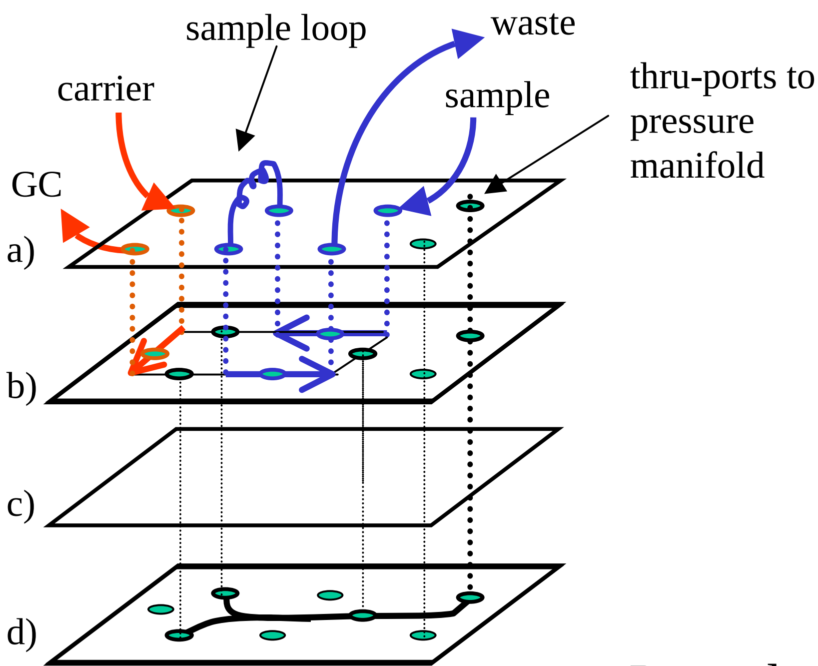
Microfabricated Diaphragm Sample Valve Design (in experimental stages)



Advantages:

- Lower production cost
- Integrates system functionality
  - smaller dead volumes
- Robust (?)

- Pyrex layer for ports
- Silicon layer with analyte conduits and valve features
- Kapton or silicon diaphragm
- Silicon layer with P-actuation conduits

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Important Project Milestones

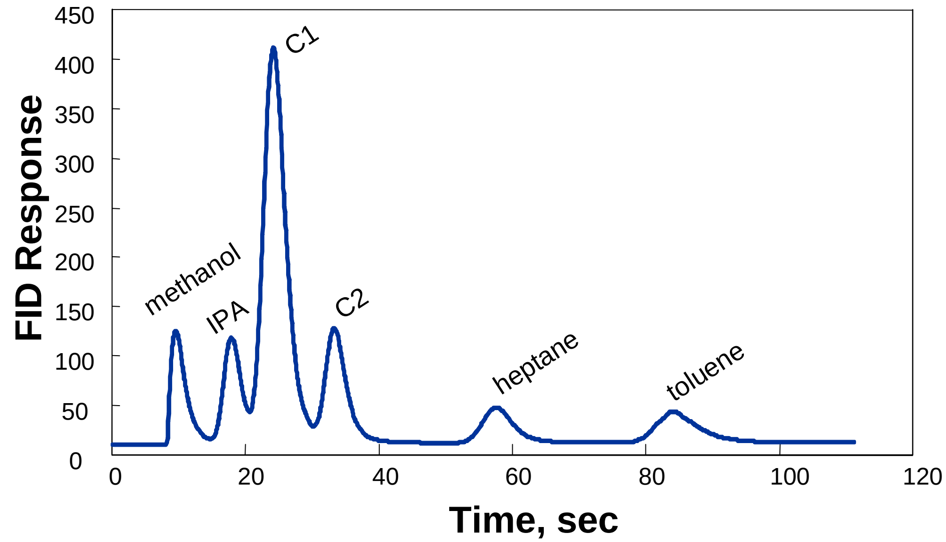
Over Project Lifetime

- Identify best column configuration - completed
- Identify best micro-detector – completed
- Design micro-gas sampling valve - completed
- Fab micro-gas sampling valve – in process
- Optimize micro-packed column – in process
- Optimize micro-PDID detector – in process
- Calibration methodology - pending
- Develop system architecture - completed
- Package components – pending
- Field test – pending
- Explore commercialization pathways – in process

Tailored Instrument Delivered to a Customer

Temperature Ramp with TEC

98 cm x 75 μm x 400 μm open μGC



Applications in fermentation, synthesis, and product drying in pharmaceutical production.

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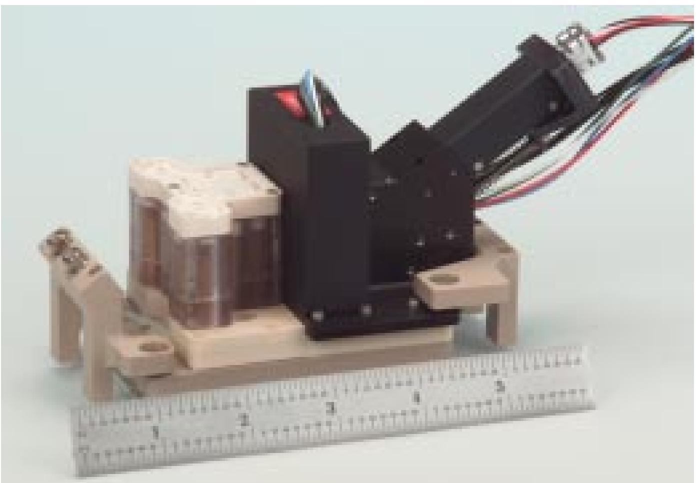
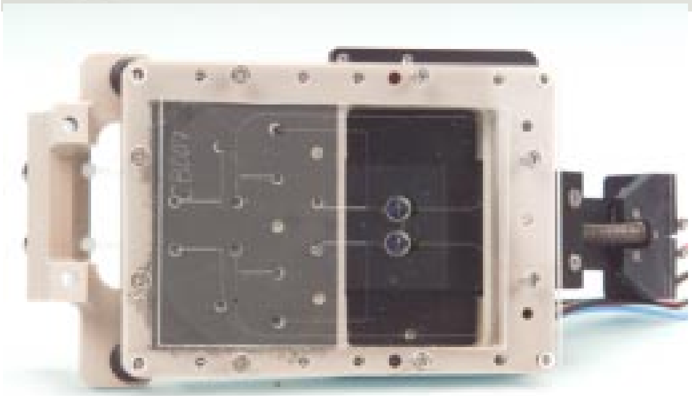
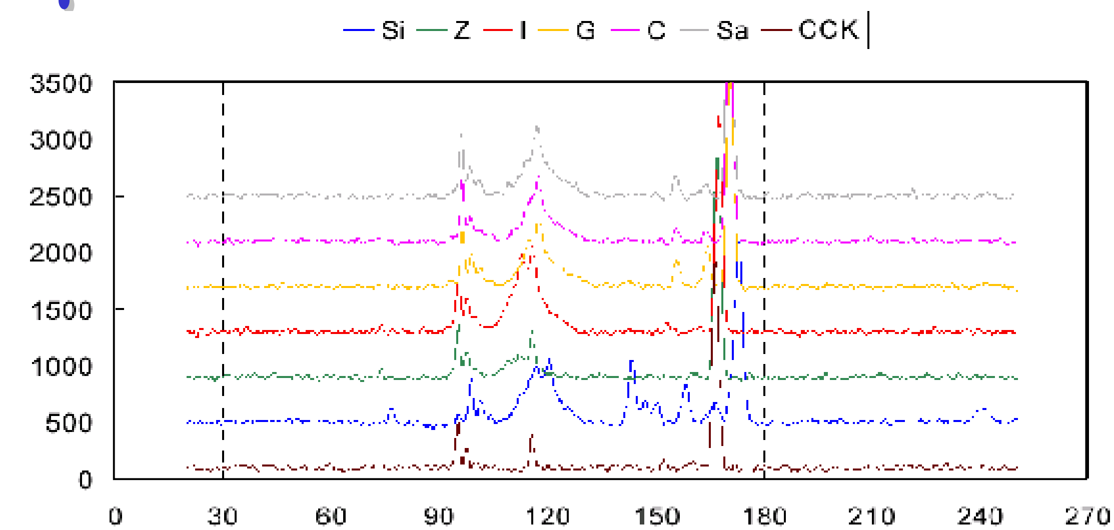
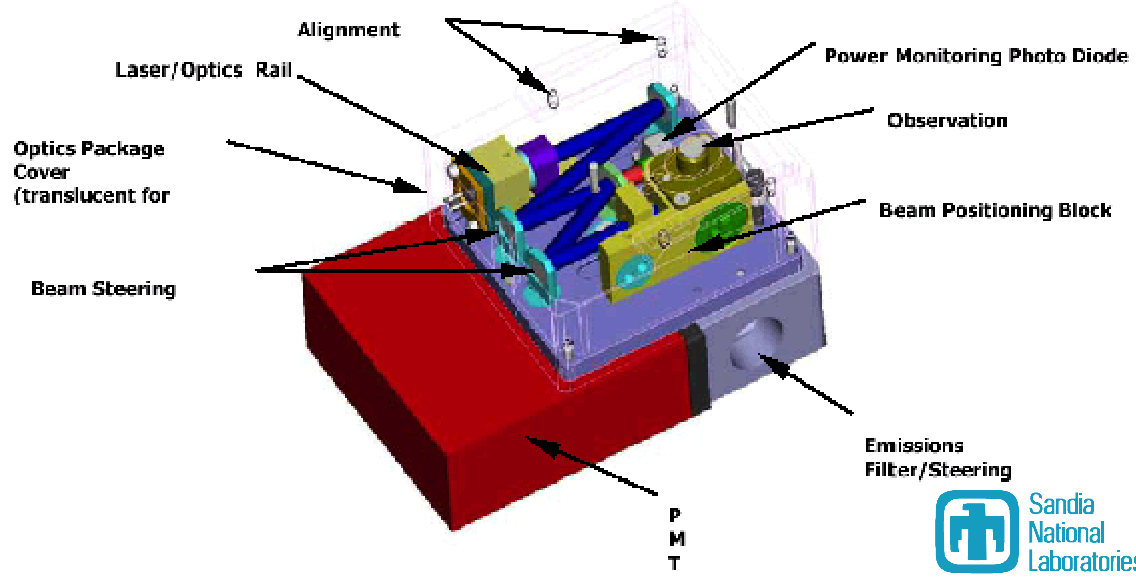
Sandia Liquid Phase μChemLab™









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Global Conclusions

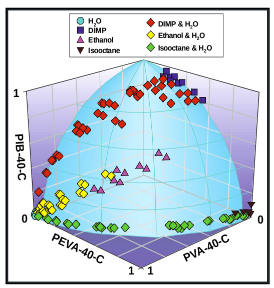
- **μChemLab systems can meet many important applications.**
- **Microfabrication provides small size, low power, rapid heating, low dead volumes, and potentially low cost.**
- **We have designed, fabricated, and demonstrated:**
  - **Rapid, low power sample preconcentrators; use of injection valves**
  - **Miniature gas chromatograph columns** (open and packed)
  - **High sensitivity sensors** (SAW, PDID, TCD)
- **Small, autonomous chemical analyzers are currently being assembled and tested for various applications**
  - **government:** military, 3-letter agencies
  - **industrial:** petroleum, pharmaceutical, energy production, others?

Short list of Sandia Sensor Technologies

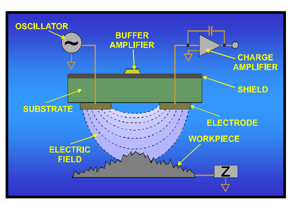
- µChemLab

- Micro Ion Mobility Spectrometer

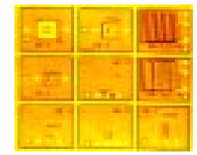
- Chemiresistor and SAW Arrays for pattern recognition



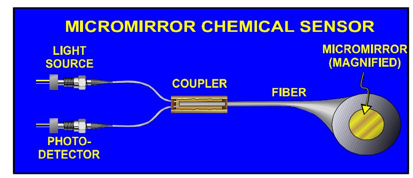
- Fringe Field Sensors



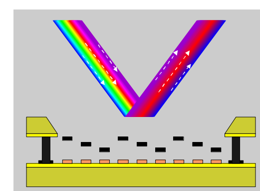
Ionizing Radiation Sensors



- Fiber Optic Chemical Sensors



- Programmable Diffraction Grating



- Combustible Gas Detector
- Hydrogen Sensors
- Portable Cloud Point Detector

In Development: µFID, µTCD